

**PCT**

WORLD INTELLECTUAL PROPERTY ORGANIZATION  
International Bureau



**INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)**

<b>(51) International Patent Classification <sup>6</sup> :</b> <b>C03C 13/00</b>	<b>A1</b>	<b>(11) International Publication Number:</b> <b>WO 96/34836</b> <b>(43) International Publication Date:</b> 7 November 1996 (07.11.96)
<b>(21) International Application Number:</b> PCT/US96/05992 <b>(22) International Filing Date:</b> 30 April 1996 (30.04.96)  <b>(30) Priority Data:</b> 08/434,223 4 May 1995 (04.05.95) US  <b>(71) Applicant:</b> OWENS CORNING [US/US]; Fiberglas Tower, Toledo, OH 43659 (US).  <b>(72) Inventors:</b> RAPP, Charles, F.; 1648 Golden Drive, Newark, OH 43055 (US). MATTSON, Stephanie, M.; 686 Earliglow Court, Newark, OH 43055 (US).  <b>(74) Agents:</b> BRUESKE, Curtis, B. et al.; Owens Corning Science & Technology Center, Building 54-1, 2790 Columbus Road, Granville, OH 43023-1200 (US).		<b>(81) Designated States:</b> AU, BR, CA, CN, JP, MX, European patent (AT, BE, CH, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE).  <b>Published</b> <i>With international search report.</i>
<b>(54) Title:</b> GLASS COMPOSITIONS AND FIBERS THEREFROM  <b>(57) Abstract</b>  Fiberizable glass composition with increased levels of BaO are suitable for insulation. These glasses have physical properties which allow current processes to fabricate them into insulation. The glasses also meet proposed German regulations regarding KI $\geq$ 40.		

**FOR THE PURPOSES OF INFORMATION ONLY**

Codes used to identify States party to the PCT on the front pages of pamphlets publishing international applications under the PCT.

AM	Armenia	GB	United Kingdom	MW	Malawi
AT	Austria	GE	Georgia	MX	Mexico
AU	Australia	GN	Guinea	NE	Niger
BB	Barbados	GR	Greece	NL	Netherlands
BE	Belgium	HU	Hungary	NO	Norway
BF	Burkina Faso	IE	Ireland	NZ	New Zealand
BG	Bulgaria	IT	Italy	PL	Poland
BJ	Benin	JP	Japan	PT	Portugal
BR	Brazil	KE	Kenya	RO	Romania
BY	Belarus	KG	Kyrgyzstan	RU	Russian Federation
CA	Canada	KP	Democratic People's Republic of Korea	SD	Sudan
CF	Central African Republic	KR	Republic of Korea	SE	Sweden
CG	Congo	KZ	Kazakhstan	SG	Singapore
CH	Switzerland	LI	Liechtenstein	SI	Slovenia
CI	Côte d'Ivoire	LK	Sri Lanka	SK	Slovakia
CM	Cameroon	LR	Liberia	SN	Senegal
CN	China	LT	Lithuania	SZ	Swaziland
CS	Czechoslovakia	LU	Luxembourg	TD	Chad
CZ	Czech Republic	LV	Latvia	TG	Togo
DE	Germany	MC	Monaco	TJ	Tajikistan
DK	Denmark	MD	Republic of Moldova	TT	Trinidad and Tobago
EE	Estonia	MG	Madagascar	UA	Ukraine
ES	Spain	ML	Mali	UG	Uganda
FI	Finland	MN	Mongolia	US	United States of America
FR	France	MR	Mauritania	UZ	Uzbekistan
GA	Gabon			VN	Viet Nam

## GLASS COMPOSITIONS AND FIBERS THEREFROM

### TECHNICAL FIELD

This invention relates to sodium barium borosilicate glass compositions.

5   Fibers from these compositions make glass fiber insulation.

### BACKGROUND ART

Glass fiber insulation is well known and has been a commercial product for a long period of time. The insulation is made from intertwined soda lime alumina borosilicate glass fiber which a binder holds together. The binder may be any suitable  
10   material but quite commonly is a phenol-formaldehyde resin or a ureaformaldehyde resin. These binders are well known, and a spray nozzle generally applies them to the glass fibers as hot gases attenuate the fibers from a rotating device, commonly called a spinner. A conveyer collects the binder-coated fibers in the form of a blanket, and heat cures the blanket to produce the final insulation. The process produces various densities by varying  
15   the conveyor speed and the thickness of the cured insulation.

The German government has proposed regulations for glass fibers. Glass fiber compositions meeting the regulations are considered to be free of suspicion. The problem, however, for the manufacturer is to produce glass fibers which meet the regulations and standard criteria. These glasses must meet the proposed regulations, be  
20   fiberizable in standard wool processes, have sufficient durability, and have acceptable insulating properties.

### DISCLOSURE OF INVENTION

We have developed glass compositions which meet all the criteria. Our glasses have a high BaO content, a high soda content, and contain small amounts of  
25   alumina. The glass compositions meet the proposed German regulations. They have a numerical index (KI) greater than or equal to 40. Standard wool processes easily produce fibers from these compositions. The difference ( $\Delta T$ ) between the temperature where the glass has a viscosity of 1,000 poise and the liquidus temperature generally is 50°F (28°C) and is often 200°F (111°C) or greater. Durability as shown by fiber weight loss is  
30   good and is often less than 5% fiber weight loss after 24 hours in water at 205°F (96°C).

**BEST MODE FOR CARRYING OUT THE INVENTION**

We made measurements of viscosity, liquidus, and durability for a number of glasses with  $KI \geq 40$ . The measurements indicated a compositional region of glasses which are useful for production of glass fiber insulation. This range is as follows for the major

5 components:

	<u>Ingredients</u>	<u>Weight Percent</u>
	$SiO_2$	45-60
	$Al_2O_3$	0-3
	$B_2O_3$	0-20
10	$Na_2O + K_2O$	12-22
	$MgO + CaO$	0-14
	$BaO$	2-35

and less than about 10% of any or all of  $ZnO$ ,  $TiO_2$ ,  $Fe_2O_3$ ,  $ZrO_2$ ,  $SrO$ , or  $Li_2O$ , the total being 100% by weight, wherein each glass composition has a KI value equal to or greater

15 than 40.

More preferably, the glass compositions consist essentially of:

	<u>Ingredients</u>	<u>Weight Percent</u>
	$SiO_2$	45-57
	$Al_2O_3$	0.8-3
20	$B_2O_3$	0-18
	$Na_2O + K_2O$	12-22
	$MgO + CaO$	0-14
	$BaO$	2-30

and less than about 5% of any or all of  $ZnO$ ,  $TiO_2$ ,  $Fe_2O_3$ ,  $ZrO_2$ ,  $SrO$ , or  $Li_2O$ , the total being 100% by weight, and wherein each glass composition has a KI value equal to or greater than 40.

Even more preferably, the glass compositions have a  $BaO$  content of 5 to 30 weight percent and less than about 2% of any or all of  $ZnO$ ,  $TiO_2$ ,  $Fe_2O_3$ ,  $ZrO_2$ ,  $SrO$ , or  $Li_2O$ . Even more preferably the glass compositions have a  $BaO$  content of 12 to 25 weight percent, and less than 2% of any or all of  $ZnO$ ,  $TiO_2$ ,  $Fe_2O_3$ ,  $ZrO_2$ ,  $SrO$ , or  $Li_2O$ .

Insulation products of these glass fibers can be produced from a conventional fiber-forming process.

The German regulation requires a numerical index (KI) greater than or equal to 40 to be considered free of suspicion. The index is calculated by  $KI = \sum (Na_2O, K_2O, CaO, MgO, BaO, B_2O_3) - 2Al_2O_3$ . This places severe restrictions on alumina levels and anything not included in the index, such as silica. The obvious choice is to lower alumina to very low levels. These glasses, however, have poor durabilities and most could not be fiberized by a standard wool process. We discovered that high BaO levels yielded glasses with significantly improved durability and fiberizability by standard processes.

We designed an array of glasses to show the effect of glass composition in this narrow field on select properties. All glasses are shown together with measured properties in the following table.

Examples of the compositions are given in the following table. All the glass compositions have KI index of 40 or higher.

Example 1

Glass fibers of the present invention have the following compositions. Most of the compositions have measured forming temperatures (the temperature where the viscosity of the glass is about 1,000 poise) and liquidus temperatures.

**TABLE**  
**Weight Percent**

<u>Ingredients</u>	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>	<u>5</u>	<u>6</u>	<u>7</u>	<u>8</u>
SiO <sub>2</sub>	54.5	55.0	56.3	56.4	56.5	56.5	56.3	56.4
5 Al <sub>2</sub> O <sub>3</sub>	0.9	1.0	1.0	1.0	1.0	0.9	1.0	1.0
B <sub>2</sub> O <sub>3</sub>	1.5	1.5	15.1	13.3	9.8	6.2	13.3	11.6
Na <sub>2</sub> O	12.4	12.5	14.5	14.4	14.1	13.8	14.6	14.5
K <sub>2</sub> O	6.0	6.0	0.8	0.8	0.7	0.7	0.8	0.8
MgO	1.1	1.1	2.6	2.6	2.5	2.5	3.1	3.0
10 CaO	3.1	3.1	7.3	7.1	7.0	6.9	8.5	8.4
BaO	17.8	17.8	2.0	4.0	8.0	12.1	2.0	4.0
TiO <sub>2</sub>	-	-	0.1	0.1	0.1	0.1	0.1	0.1
Fe <sub>2</sub> O <sub>3</sub>	0.2	0.2	0.3	0.3	0.3	0.3	0.3	0.3
ZrO <sub>2</sub>	2.5	1.8	-	-	-	-	-	-
15 Temperature °F (°C)	1903	1882	1737	1738	1761	1789	1739	1747
for Viscosity of	(1039)	(1028)	(947)	(948)	(961)	(976)	(948)	(953)
1,000 Poise								
Liquidus	1524	-	1498	1481	1548	1553	1578	1567
Temperature °F (°C)	(829)	(-)	(814)	(805)	(842)	(845)	(859)	(853)
20 KI	40.0	40.0	40.3	40.3	40.3	40.3	40.3	40.3
Durability	7.0	-	4.7	4.3	3.8	3.6	-	-
(% Wt Loss of								
Fibers After								
24 Hours)*								
25 *Exposure to water at 205°F (96°C)								

<u>Ingredients</u>		<u>9</u>	<u>10</u>	<u>11</u>	<u>12</u>
	SiO <sub>2</sub>	54.5	54.4	55.2	54.8
	Al <sub>2</sub> O <sub>3</sub>	1.0	1.0	1.6	1.7
	B <sub>2</sub> O <sub>3</sub>	15.2	15.4	-	-
5	Na <sub>2</sub> O	14.5	14.4	15.5	16.5
	K <sub>2</sub> O	0.8	0.8	-	-
	MgO	2.6	2.6	-	1.8
	CaO	7.2	7.2	-	4.0
	BaO	2.0	2.0	27.7	21.2
10	TiO <sub>2</sub>	2.0	0.1	-	-
	Fe <sub>2</sub> O <sub>3</sub>	0.3	0.3	-	-
	ZrO <sub>2</sub>	-	2.0	-	-
	Temperature °F (°C) for Viscosity of	1714 (934)	1740 (949)	1882 (1028)	1867 (1019)
15	1,000 Poise				
	Liquidus	1538	1551	-	-
	Temperature °F (°C)	(837)	(844)	(-)	(-)
	KI	40.3	40.3	40.0	40.0
	Durability	-	-	-	-
20	(% Wt Loss of Fibers After 24 Hours)*				
	*Exposure to water at 205°F (96°C)				

25

30

CLAIMS

1. Fiberizable glass compositions suitable for insulation consisting essentially of:

	<u>Ingredients</u>	<u>Weight Percent</u>
5	SiO <sub>2</sub>	45-60
	Al <sub>2</sub> O <sub>3</sub>	0-3
	B <sub>2</sub> O <sub>3</sub>	0-20
	Na <sub>2</sub> O + K <sub>2</sub> O	12-22
	MgO + CaO	0-14
10	BaO	2-35

and less than about 10% of any or all of ZnO, TiO<sub>2</sub>, Fe<sub>2</sub>O<sub>3</sub>, ZrO<sub>2</sub>, SrO, or Li<sub>2</sub>O, the total being 100% by weight, and wherein each glass composition as a KI value equal to or greater than 40.

2. Fiberizable glass compositions suitable for insulation consisting essentially of:

	<u>Ingredients</u>	<u>Weight Percent</u>
	SiO <sub>2</sub>	47-57
	Al <sub>2</sub> O <sub>3</sub>	0.8-3
	B <sub>2</sub> O <sub>3</sub>	0-18
20	Na <sub>2</sub> O + K <sub>2</sub> O	12-22
	MgO + CaO	0-14
	BaO	2-30

and less than about 5% of any or all of ZnO, TiO<sub>2</sub>, Fe<sub>2</sub>O<sub>3</sub>, ZrO<sub>2</sub>, SrO, or Li<sub>2</sub>O, the total being 100% by weight, and wherein each glass composition as a KI value equal to or greater than 40.

3. Glass compositions according to claim 2 having a BaO content of 5 to 30 weight percent and less than about 2% of any or all of ZnO, TiO<sub>2</sub>, Fe<sub>2</sub>O<sub>3</sub>, ZrO<sub>2</sub>, SrO, or Li<sub>2</sub>O.

4. Glass compositions according to claim 2 having a BaO content of 12 to 25 weight percent and less than about 2% of any or all of ZnO, TiO<sub>2</sub>, Fe<sub>2</sub>O<sub>3</sub>, ZrO<sub>2</sub>, SrO, or Li<sub>2</sub>O.



5. Glass compositions according to claim 1 having the compositions of:

	<u>Ingredients</u>	<u>Weight Percent</u>
	SiO <sub>2</sub>	54.5
	Al <sub>2</sub> O <sub>3</sub>	0.9
5	B <sub>2</sub> O <sub>3</sub>	1.5
	Na <sub>2</sub> O	12.4
	K <sub>2</sub> O	6.0
	MgO	1.1
	CaO	3.1
10	BaO	17.8
	Fe <sub>2</sub> O <sub>3</sub>	0.2
	ZrO <sub>2</sub>	2.5

6. Glass fibers having glass compositions according to any one of claims

1 to 5.

15

7. Glass fiber insulation comprising a collection of glass fibers according

to claim 6.

20

25

30

# INTERNATIONAL SEARCH REPORT

Int. Application No  
PCT/US 96/05992

**A. CLASSIFICATION OF SUBJECT MATTER**  
IPC 6 C03C13/00

According to International Patent Classification (IPC) or to both national classification and IPC

**B. FIELDS SEARCHED**

Minimum documentation searched (classification system followed by classification symbols)  
IPC 6 C03C

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

**C. DOCUMENTS CONSIDERED TO BE RELEVANT**

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US,A,5 055 428 (R.M. PORTER) 8 October 1991 see claim 1 ---	1,2,6
X	EP,A,0 019 600 (OY PARTEC AB) 26 November 1980 see claim 1 ---	1,2,6
X	CHEMICAL ABSTRACTS, vol. 99, no. 12, 19 September 1983 Columbus, Ohio, US; abstract no. 92677w, page 281; XP002009885 see abstract & JP,A,05 860 641 (NIPPON ELECTRIC GLASS CO. LTD.) 11 April 1983 --- -/-	1-3,6

☒ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

\* Special categories of cited documents:

- \*A\* document defining the general state of the art which is not considered to be of particular relevance
- \*E\* earlier document but published on or after the international filing date
- \*L\* document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
- \*O\* document referring to an oral disclosure, use, exhibition or other means
- \*P\* document published prior to the international filing date but later than the priority date claimed

- \*T\* later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
- \*X\* document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
- \*Y\* document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.
- \*Z\* document member of the same patent family

Date of the actual completion of the international search

31 July 1996

Date of mailing of the international search report

19.08.96

Name and mailing address of the ISA

European Patent Office, P.B. 5818 Patentlaan 2  
NL - 2280 HV Rijswijk  
Tel. (+31-70) 340-2040, Tx. 31 651 epo nl,  
Fax: (+31-70) 340-3016

Authorized officer

Reedijk, A

# INTERNATIONAL SEARCH REPORT

International Application No  
PCT/US 96/05992

## C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
P,X	WO,A,95 32925 (ISOVER SAINT GOBAIN) 7 December 1995 see abstract ---	1-3,6,7
X	US,A,4 289 518 (J.A. BATTIGELLI ET AL.) 15 September 1981 see table 2 ---	1-3,6,7
A	US,A,2 335 463 (H. STEINBOCK) 30 November 1943 see the whole document -----	1-7

# INTERNATIONAL SEARCH REPORT

information on patent family members

Int. Application No

PCT/US 96/05992

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US-A-5055428	08-10-91	AU-B- 8625091	15-04-92
		CA-A- 2071511	27-03-92
		DE-D- 69109083	24-05-95
		DE-T- 69109083	31-08-95
		EP-A- 0502159	09-09-92
		ES-T- 2072016	01-07-95
		JP-T- 5502432	28-04-93
		WO-A- 9205121	02-04-92
EP-A-19600	26-11-80	SE-B- 418961	06-06-81
		JP-C- 1195154	12-03-84
		JP-A- 56014450	12-02-81
		JP-B- 58024385	20-05-83
		SE-A- 7904044	10-11-80
		US-A- 4312952	26-01-82
		US-A- 4381347	26-04-83
WO-A-9532925	07-12-95	DE-A- 4418727	22-02-96
		AU-B- 2616295	21-12-95
		BR-A- 9506253	16-04-96
		EP-A- 0711256	15-05-96
		FI-A- 960105	18-01-96
		NO-A- 960336	26-01-96
US-A-4289518	15-09-81	FR-A- 2443436	04-07-80
		AR-A- 219020	15-07-80
		AR-A- 218178	15-05-80
		AR-A- 218794	30-06-80
		AU-B- 534657	09-02-84
		AU-B- 5364779	12-06-80
		AU-B- 534642	09-02-84
		AU-B- 5364879	12-06-80
		AU-B- 536496	10-05-84
		AU-B- 5364979	28-01-82
		BE-A- 880485	06-06-80
		BG-A- 41654	15-07-87
		BG-A- 41655	15-07-87
		BG-A- 41656	15-07-87
		CA-A- 1129202	10-08-82
		CA-A- 1129203	10-08-82

# INTERNATIONAL SEARCH REPORT

information on patent family members

International Application No

PCT/US 96/05992

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US-A-4289518		CA-A- 1129204	10-08-82
		CA-A- 1128757	03-08-82
		CA-A- 1136165	23-11-82
		CA-A- 1136418	30-11-82
		CA-A- 1152329	23-08-83
		CH-A- 634027	14-01-83
		CH-A- 633763	31-12-82
		CH-A- 643219	30-05-84
		DE-A- 2911510	19-06-80
		DE-C- 2954306	29-10-87
		DE-C- 2954307	28-08-86
		DE-A- 2954455	04-07-85
		GB-A,B 2041910	17-09-80
		GB-A,B 2041911	17-09-80
		GB-A,B 2045228	29-10-80
		JP-C- 1412292	27-11-87
		JP-A- 55113637	02-09-80
		JP-B- 62020141	06-05-87
		JP-C- 1412293	27-11-87
		JP-A- 55113638	02-09-80
		JP-B- 62020142	06-05-87
		JP-C- 1432182	24-03-88
		JP-A- 55113646	02-09-80
		JP-B- 62042869	10-09-87
		JP-C- 1456920	09-09-88
		JP-A- 61141637	28-06-86
		JP-B- 63005337	03-02-88
		JP-C- 1456921	09-09-88
		JP-A- 61141638	28-06-86
		JP-B- 63005336	03-02-88
		LU-A- 81962	01-07-80
		NL-A- 7908864	10-06-80
		NL-A- 7908865	10-06-80
		NL-A- 7908866	10-06-80
-----			
US-A-2335463	30-11-43	NONE	
-----			